

FORM-PTO-1390
(Rev. 9-2001)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

022701-969

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.8)

UNASSIGNED 09/980785

INTERNATIONAL APPLICATION NO.
PCT/FR00/01543

INTERNATIONAL FILING DATE
6 JUNE 2000

PRIORITY DATE CLAIMED
7 JUNE 1999

TITLE OF INVENTION

USE OF POLYALKOXYLATED TERPENE DERIVATIVES FOR TREATING TEXTILE FIBRES

APPLICANT(S) FOR DO/EO/US

Gilles LORENTZ and André VAN DER SPUY

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☒ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

International Preliminary Examination Report (Form PCT/IPEA/409; Notification from the IB of WIPO re: Designated Offices (Form PCT/IB/308); Official Notification of Election from the IB (Form PCT/IB/332).



21839

U.S. APPLICATION NO. (if known) 37 C.F.R. 1.1(a) UNASSIGNED		INTERNATIONAL APPLICATION NO. PCT/FR00/01543		ATTORNEY'S DOCKET NUMBER 022701-969	
21. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)):					
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report prepared by the EPO or JPO \$1,040.00 (960)					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$890.00 (970)					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00 (958)					
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00 (956)					
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 (962)					
ENTER APPROPRIATE BASIC FEE AMOUNT =					
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.432(a)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>					
Claims	Number Filed	Number Extra	Rate		
Total Claims	14 -20 =	0	X\$18.00 (966)	\$	0.00
Independent Claims	1 -3 =	0	X\$84.00 (964)	\$	0.00
Multiple dependent claim(s) (if applicable)			+ \$280.00 (968)	\$	0.00
TOTAL OF ABOVE CALCULATIONS =				\$	890.00
Reduction for 1/2 for filing by small entity, if applicable (see below).				+	\$ -
SUBTOTAL =				\$	890.00
Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(h)).				+	
TOTAL NATIONAL FEE =				\$	890.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property				+	
TOTAL FEES ENCLOSED =				\$	890.00
				Amount to be refunded:	\$
				charged:	\$
<p>a. <input type="checkbox"/> Small entity status is hereby claimed.</p> <p>b. <input checked="" type="checkbox"/> A check in the amount of \$ <u>890.00</u> to cover the above fees is enclosed.</p> <p>c. <input type="checkbox"/> Please charge my Deposit Account No. <u>02-4800</u> in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>d. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>02-4800</u>. A duplicate copy of this sheet is enclosed.</p>					
<p>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p>					
SEND ALL CORRESPONDENCE TO:					
Norman H. Stegno BURNS, DOANE, SWECKER & MATHIS, L.L.P. P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620			SIGNATURE TERESA STANEK REA NAME 30,427 REGISTRATION NUMBER		
			DECEMBER 6, 2001 DATE		

Patent

Attorney's Docket No. 022701-969

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)	
)	
Gilles LORENTZ <i>et al.</i>)	Group Art Unit: Unassigned
)	
Application No.: UNASSIGNED)	Examiner: Unassigned
(Corresponds to PCT/FR00/01543))	
)	
International Filing Date: 6 JUNE 2000)	
)	
For: USE OF POLYALKOXYLATED)	
TERPENE DERIVATIVES IN)	
TREATING TEXTILE FIBRES)	
)	

PRELIMINARY AMENDMENT**BOX PCT**

Assistant Commissioner for Patents
Washington, D.C. 20231

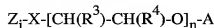
Sir:

Prior to examination, please amend the above-captioned application as follows:

IN THE CLAIMS:

Kindly amend claims 1-9 as follows:

1. (Amended) A process for treating textile fibres comprising at least one of the following steps: a) singeing, b) desizing, c) cleaning, d) bleaching, e) mercerisation, f) dyeing steps, and g) finishing, wherein a compound with formula (I) is used in at least one of the above steps:



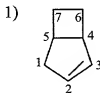
where

- Z_1 represents a bicyclo[a,b,c]heptenyl radical or a bicyclo[a,b,c]heptyl radical, optionally substituted by at least one C_1-C_6 alkyl radical;

a, b and c being such that

- $a+b+c=5$;
- $a=2, 3$ or 4 ;
- $b=1$ or 2 ;
- $c=0$ or 1 ;

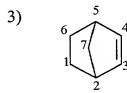
and comprising a skeleton selected from those shown below (Z_1 to Z_7), and from their corresponding heptyls with no double bond:



[3.2.0]



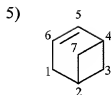
[3.2.0]



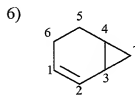
[2.2.1]



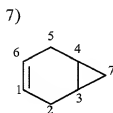
[3.1.1]



[3.1.1]



[4.1.0]



[4.1.0]

- X represents $\text{-CH}_2\text{-C(R}^1\text{)(R}^2\text{)-O-}$ or $\text{-O-CH(R}^1\text{)-CH(R}^2\text{)-O-}$ in which:
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_6$ hydrocarbon radical;
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical;
- R^3 and R^4 , which may be identical or different, represent a hydrogen atom or a linear or branched $\text{C}_1\text{-C}_{22}$ (cyclo)alkyl or (cyclo)alkenyl group;
- n is an average value in the range 1 to 200;
- A represents a hydrogen atom, a $\text{C}_1\text{-C}_6$ alkyl radical, an aryl or alkylaryl radical, a halogen atom, a $\text{-CH}_2\text{-CH(OH)R}^5$ group where R_5 represents a

linear or branched or cyclic C_1 - C_{22} alkyl radical or an aryl radical, or a group selected from $-SO_3M$, $-OPO_3(M)_2$, $-(CH_2)_a-COOM$, $-(CH_2)_b-SO_3M$, where a and b are from 1 to 6, M representing H, Na, K, Li, $N(R_4)^+$ where radicals R, which may be identical or different, represent a hydrogen atom or a linear or branched or cyclic C_1 - C_{22} alkyl radical which may be hydroxylated;

or a mixture of these compounds.

2. (Amended) A process according to claim 1, wherein a compound with formula (I) is used wherein radical Z_1 is attached to the chain residue via any one of its carbon atoms 1 to 6.

3. (Amended) A process according to claim 1, wherein the compound is such that in formula (I), radical Z is substituted by two C_1 - C_6 alkyl groups, on at least one of its carbon atoms.

4. (Amended) A process according to claim 1, wherein a compound with formula (I) is used wherein X represents $-CH_2-C(R^1)(R^2)-O-$ and radicals Z_i correspond to radicals Z_3 to Z_7 .

5. (Amended) A process according to claim 1, wherein a compound with formula (I) is used in which X represents $-O-CH(R'^1)-CH(R'^2)-O-$, radical Z_1 corresponds to radical Z_3 not comprising an ethylenically unsaturated bond.

6. (Amended) A process according to claim 1, wherein the compound is such that in formula (I), radical Z is substituted by a C_1-C_6 alkyl radical.

7. (Amended) A process according to claim 1, wherein a mixture of compounds with formula (I) is used in which at least one thereof is such that the group $--[CH(R^3)-CH(R^4)-O]-$ corresponds to the following sequence: $--[CH(R^3)-CH(R^4)-O]_p-[CH_2-CH_2-O]_q-[CH(R^3)-CH(R^4)-O]_r-$, where R^3 or R^4 is other than hydrogen, and $n=p+q+r$.

8. (Amended) A process according to claim 1, wherein the compound with formula (I) is used during dyeing steps e).

9. (Amended) A process according to claim 1, wherein the compound with formula (I) is used in a concentration in the range 1 to 5 g/l.

Kindly add the following new claims 10-14:

- 10. The process according to claim 1, wherein Z_1 represents a bicyclo[a,b,c]heptenyl radical or a bicyclo[a,b,c]heptyl radical, optionally substituted by a methyl radical.
11. The process according to claim 2, wherein Z_1 is attached to the chain residue via carbon atoms 1,5 or 6.
12. The process according to claim 3, wherein radical Z is substituted by two C_1 - C_6 alkyl groups on carbon 7.
13. The process according to claim 4, wherein Z_1 corresponds to radicals Z_4 or Z_5 .
14. The process according to claim 6, wherein the radical Z is substituted by a methyl radical on carbon 2 or 5 of the bi-cycle.--

REMARKS

Entry of the foregoing amendments are respectfully requested.

Should the Examiner have any questions concerning the subject application, a telephone call to the undersigned would be appreciated.

Respectfully submitted,

BURNS, DOANE, SWICKER & MATHIS, L.L.P.

By: _____

Teresa Stanek Rea
Registration No. 30,427

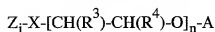
P.O. Box 1404
Alexandria, Virginia 22313-1404
(703) 836-6620

Date: December 6, 2001

Attachment to Preliminary Amendment dated December 6, 2001

Marked-up Claims 1-9

1. (Amended) A process for treating textile fibres comprising at least one of the following steps: a) singeing, b) desizing, c) cleaning, d) bleaching, e) mercerisation, f) dyeing steps, and g) finishing, [characterized in that] wherein a compound with formula (I) is used in at least one of the above steps:



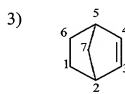
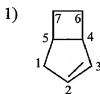
where

- Z_1 represents a bicyclo[a,b,c]heptenyl radical or a bicyclo[a,b,c]heptyl radical, optionally substituted by at least one C_1-C_6 alkyl radical[, preferably methyl];

a, b and c being such that

- $a+b+c=5$;
- $a=2, 3$ or 4 ;
- $b=1$ or 2 ;
- $c=0$ or 1 ;

and comprising a skeleton selected from those shown below (Z_1 to Z_7), and from their corresponding heptyls with no double bond:

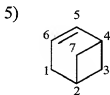


Attachment to Preliminary Amendment dated December 6, 2001

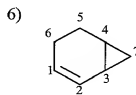
Marked-up Claims 1-9



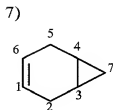
[3.1.1]



[3.1.1]



[4.1.0]



[4.1.0]

- X represents $\text{-CH}_2\text{-C(R}^1\text{)(R}^2\text{)-O-}$ or $\text{-O-CH(R}^1\text{)-CH(R}^2\text{)-O-}$ in which:
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_6$ hydrocarbon radical;
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical;
- R^3 and R^4 , which may be identical or different, represent a hydrogen atom or a linear or branched $\text{C}_1\text{-C}_{22}$ (cyclo)alkyl or (cyclo)alkenyl group;
- n is an average value in the range 1 to 200;

Attachment to Preliminary Amendment dated December 6, 2001

Marked-up Claims 1-9

- A represents a hydrogen atom, a C₁-C₆ alkyl radical, an aryl or alkylaryl radical, a halogen atom, a -CH₂-CH(OH)R⁵ group where R₅ represents a linear or branched or cyclic C₁-C₂₂ alkyl radical or an aryl radical, or a group selected from -SO₃M, -OPO₃(M)₂, -(CH₂)_a-COOM, -(CH₂)_b-SO₃M, where a and b are from 1 to 6, M representing H, Na, K, Li, N(R₄)⁺ where radicals R, which may be identical or different, represent a hydrogen atom or a linear or branched or cyclic C₁-C₂₂ alkyl radical which may be hydroxylated;
or a mixture of these compounds.

2. (Amended) A process according to [the preceding claims] claim 1, [characterized in that] wherein a compound with formula (I) is used wherein radical Z₁ is [preferably] attached to the chain residue via any one of its carbon atoms 1 to 6[, and more particularly via carbon atoms 1, 5 and 6].

3. (Amended) A process according to [any one of the preceding claims] claim 1, [characterized in that] wherein the compound is such that in formula (I), radical Z is substituted by two C₁-C₆ alkyl groups, on at least one of its carbon atoms[, more particularly on carbon atom 7].

Attachment to Preliminary Amendment dated December 6, 2001

Marked-up Claims 1-9

4. (Amended) A process according to [any one of the preceding claims] claim 1, [characterized in that] wherein a compound with formula (I) is used wherein X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ and radicals Z_1 correspond to radicals Z_3 to Z_7 [, preferably to radicals Z_4 and Z_5].
5. (Amended) A process according to [any one of the preceding claims] claim 1, [characterized in that] wherein a compound with formula (I) is used in which X represents $-\text{O}-\text{CH}(\text{R}^1)-\text{CH}(\text{R}^2)-\text{O}-$, radical Z_1 corresponds to radical Z_3 not comprising an ethylenically unsaturated bond.
6. (Amended) A process according to [the preceding claims] claim 1, [characterized in that] wherein the compound is such that in formula (I), radical Z is substituted by a C_1-C_6 alkyl radical[, preferably a methyl radical on carbon 2 or 5 of the bi-cycle].
7. (Amended) A process according to [any one of the preceding claims] claim 1, [characterized in that] wherein a mixture of compounds with formula (I) is used in which at least one thereof is such that the group $-\text{CH}(\text{R}^3)-\text{CH}(\text{R}^4)-\text{O}-$ corresponds to the following sequence: $-\text{CH}(\text{R}^3)-\text{CH}(\text{R}^4)-\text{O}-\text{P}-[\text{CH}_2-\text{CH}_2-\text{O}]_q-[\text{CH}(\text{R}^3)-\text{CH}(\text{R}^4)-\text{O}]_r$, where R^3 or R^4 is other than hydrogen, and $n=p+q+r$.

Attachment to Preliminary Amendment dated December 6, 2001

Marked-up Claims 1-9

8. (Amended) A process according to [any one of the preceding claims] claim
1, [characterized in that] wherein the compound with formula (I) is used during dyeing
steps e).

9. (Amended) A process according to [any one of the preceding claims] claim
1, [characterized in that] wherein the compound with formula (I) is used in a concentration
in the range 1 to 5 g/l.

USE OF POLYALKOXYLATED TERPENE DERIVATIVES IN TREATING TEXTILE FIBRES

The present invention relates to the use, in treating textile fibres, of polyalkoxylated terpene compounds, more particularly for dyeing operations.

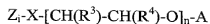
It should first be pointed out that in the following text, reference will only be made to textile fibres, as this term designates fibres, yarns, knitted fabrics, woven fabrics and non woven fabrics.

Further, the present invention is applicable to treating natural textile fibres (natural or regenerated cellulose fibres, (cotton), wool), and synthetic fibres (polyester, polyamide (nylon), polyacrylonitrile (acrylic).

The treatment of textile fibres comprises a variety of steps, including singeing, desizing, cleaning, bleaching, mercerisation, dyeing steps, and finishing. During those steps, wetting and/or penetrating agents have to be used to render the agent used effective during the step under consideration. Ethoxylated nonylphenols are normally used. Those agents are highly effective, but have the disadvantage of producing a large quantity of foam. It is possible to use them combined with an anti-foaming agent such as silicone emulsion-based anti-foaming agents. Unfortunately, under very severe shear conditions, which can be encountered during dyeing operations, the anti-foaming agents are often destabilised and a silicone gel is formed which is deposited on the fibres, causing the appearance of permanent spots on the fabric.

The present invention thus pertains to the use of a wetting/penetrating agent which is as effective as the compounds normally used in textile fibre treatment steps, but which do not have the disadvantages associated with the appearance of foam.

Thus the present invention provides a process for treating textile fibres comprising at least one of the following steps: a) singeing, b) desizing, c) cleaning, d) bleaching, e) mercerisation, f) dyeing steps, g) finishing, in which a compound with formula (I) is used in at least one of the above steps:



where

- Z_1 represents a bicyclo[a,b,c]heptenyl radical or a bicyclo[a,b,c]heptyl radical, optionally substituted by at least one C_1 - C_6 alkyl radical, preferably methyl;

a, b and c being such that

- $a + b + c = 5$;
- $a = 2, 3$ or 4 ;
- $b = 1$ or 2 ;
- $c = 0$ or 1 ;

and comprising a skeleton selected from those shown below (Z_1 to Z_7), and from their corresponding heptyls with no double bond:

1)



[3.2.0]

2)



[3.2.0]

3)



[2.2.1]

4)



[3.1.1]

5)



[3.1.1]

6)



[4.1.0]

7)



[4.1.0]

- X represents $-CH_2-C(R^1)(R^2)-O-$ or $-O-CH(R^1)-CH(R^2)-O-$ in which:
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1 - C_6 hydrocarbon radical;

- R^{11} and R^{12} , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1 - C_{22} hydrocarbon radical;
- R^3 and R^4 , which may be identical or different, represent a hydrogen atom or a linear or branched C_1 - C_{22} (cyclo)alkyl or (cyclo)alkenyl group;
- n is an average value in the range 1 to 200;
- A represents a hydrogen atom, a C_1 - C_6 alkyl radical, an aryl or alkylaryl radical, a halogen atom, a $-CH_2-CH(OH)R^5$ group where R^5 represents a linear or branched or cyclic C_1 - C_{22} alkyl radical or an aryl radical, or a group selected from $-SO_3M$, $-OPO_3(M)_2$, $-(CH_2)_a-COOM$, $-(CH_2)_b-SO_3M$, where a and b are from 1 to 6, M representing H, Na, K, Li, $N(R_4)^+$ where radicals R, which may be identical or different, represent a hydrogen atom or a linear or branched or cyclic C_1 - C_{22} alkyl radical which may be hydroxylated;

or a mixture of these compounds.

The compounds used (termed terpene compounds) will now be described.

As indicated above, the terpene compound derivatives used in the process of the invention have the formula mentioned above.

In this formula, radical Z_1 is preferably attached to the chain residue via any one of carbon atoms 1 to 6. In a more particular implementation, radical Z_1 is attached to the chain residue via carbon atoms 1, 5 and 6.

In an advantageous variation of the invention, the compounds are such that radical Z_1 is substituted in at least one of its carbon atoms by at least two C_1 - C_6 alkyl groups, preferably by two methyl radicals. More particularly, the substituents are localised on carbon atom 7.

A first family of compounds corresponds to that in which X represents $-CH_2-C(R^1)(R^2)-O-$. In this case, radicals Z_1 more particularly correspond to radicals Z_3 to Z_7 . Further, the cycles

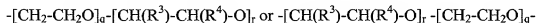
comprise an ethylenically unsaturated bond. Further, they carry two substituents, preferably methyl, on carbon atom 7. Preferred compounds are defined by radicals Z₄ and Z₅, carrying two methyl groups on carbon atom 7.

A second family of compounds corresponds to that where X represents -O-CH(R¹)-CH(R²)-O-. More particularly, the cycles contain no ethylenically unsaturated bond and are represented by radical Z₃. It should be noted that in the case of this family, at least R¹ or R² is not hydrogen.

Thus in a preferred implementation, the compounds are such that R¹ or R² is a methyl radical. In a particular characteristic of this family of compounds, carbon atom 7 carries two methyl substituents and one C₁-C₆ alkyl substituent, preferably a methyl, situated on carbon atom 2 or carbon atom 5.

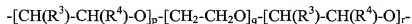
Regarding the -[CH(R³)-CH(R⁴)-O]- motifs, R³ and R⁴ have already been defined as being identical or different, representing a hydrogen atom or a linear or branched C₁-C₂₂ (cyclo)alkyl or (cyclo)alkenyl group, preferably C₁-C₆. More particularly, radicals R³ and R⁴, which may be identical or different, represent a hydrogen atom, a methyl radical or an ethyl radical.

If motifs -[CH(R³)-CH(R⁴)-O]- are different, their distribution is statistical or blocked. In this latter possibility, two or more different blocks can be envisaged. As an example, suitable compounds can contain the following sequences:



where R³ or R⁴ is other than hydrogen, and n = q + r.

One type of block compound which is suitable for the invention is that with the following sequence:



where R³ or R⁴ is other than hydrogen, and n = p + q + r.

In a particular implementation, said motifs correspond to oxyethylenated and/or oxypropylenated motifs.

n is an average value in the range 1 to 200. It corresponds to the total sum of $-\text{CH}(\text{R}^3)-\text{CH}(\text{R}^4)-\text{O}-$ motifs.

Preferably, the number of oxyethylenated motifs is in the range 1 to 200, more preferably in the range 1 to 50, more particularly in the range 1 to 20.

Regarding the number of oxypropylenated motifs, their average number is in the range 0 to 20, more particularly in the range 1 to 10, preferably in the range 1 to 4.

It may be highly advantageous to use a terpene compound corresponding to a mixture of compounds with formula (I) wherein at least one thereof is such that it has the block mentioned above.

These compounds, and a method for their preparation have been described in International patent applications WO-A-96/01245 and WO-A-98/28249. Reference should be made thereto for a definition and the production of those compounds.

The process of the invention thus consists of using the compounds described above in one or more steps carried out when treating textile fibres.

It has been established that said compounds have important textile fibre wetting properties encouraging penetration of the reactant under consideration into the textile fibres.

Further, the compounds used in the invention have good resistance under the often severe conditions encountered during fibre treatment. Many steps are carried out in highly alkaline media.

Further, the compounds of the invention have the advantage of adapting their structure to the demands of the operation in which they are used. Thus using a common base, it is possible to use a non ionic or ionic effective compound, compatible with the various compounds present in the solution intended for the fibre treatment.

The textile fibres which can be treated contain residual additives from previous steps, such as lubricants, softening additives (lubricants for dies in the case of synthetic fibres), polyvinyl alcohol, carboxymethylcellulose, fibre cohesion agents (starch), film-forming agents (hydrocolloids). These compounds have to be removed from the fibres. A desizing (step b)) is used to carry out this operation.

The desizing operation can be carried out enzymatically (to depolymerise starch, if present), and/or by chemical oxidation and/or by alkali treatment. More particularly, the oxidation reaction is carried out with an oxidising agent selected from hydrogen peroxide, persulphates, or perborates, for example. It is also carried out in an alkaline medium, such as alkali treatment. Hydroxides of alkali metals (such as sodium hydroxide) and alkali metal carbonates and bicarbonates can be used; sodium hydroxide is preferred. Normally, the concentration of alkaline agent is of the order of 2 to 5 g/l, to obtain a pH in the range 10 to 12. The desizing operation can also comprise the usual additives (chelating agents, etc.).

This step is carried out in solution comprising 70% to 80% of water.

Further, it is carried out hot. Thus temperatures of more than 50°C are routine.

Further, desizing can be carried out in the presence of the terpene compound described, the use of which forms the subject matter of the invention.

The amount of this compound in the desizing liquor is more particularly in the range 1 to 5 g/l.

In general, the textile fibres are impregnated in the solution, either continuously (steam box) or batchwise (rotating rollers).

This step can be carried out in one or more passes.

Depending on the nature of the textile fibre, it may be necessary or simply advantageous to carry out a preliminary step known as singeing (step a)). This step provides the textile fibre with a smoother surface. It consists of burning the fibres which protrude from the surface.

It should be noted that carrying out this singeing step does not facilitate the desizing step. The textile fibre which has undergone such a treatment is actually overdry and as a result a solution with a strong wetting power is imperative if the efficiency of the desizing step is to be retained.

Once the desizing step has been carried out, a cleaning step (step c)) can be carried out. This step is more generally used when the textile fibres include cotton. This operation aims to eliminate the compounds naturally present in cotton fibres, such as cellulose residues (hemicellulose, immature cellulose), or natural oils or waxes which cause heterogeneities in the final, dyed or non dyed fabric.

This cleaning operation is carried out in a highly alkaline medium.

The concentration of the alkaline agent in the liquor is normally 20 to 40 g/l.

Advantageously, sodium hydroxide is used.

The medium can also comprise the terpene compound. The amount of this compound is advantageously in the range 1 to 5 g/l.

More particularly, the step is carried out at a temperature of the order of 60-80°.

This step is generally carried out in a steam box.

At the end of this operation, the textile fibres can be directly dyed provided that the colours are dark.

However, the textile fibre treatment process is normally continued by a bleaching step (step d)).

The operation is carried out using an oxidising agent, more particularly hydrogen peroxide.

It may be advantageous to use a stabilising agent such as a silicate, hypochlorite or polyphosphoric acid.

The bleaching step is carried out at high temperatures, of the order of 70°C to 80°C.

At the end of the bleaching step, a mercerisation or slack mercerisation step (step d)) can optionally be carried out.

This step consists of immersing the textile fibres in an alkaline solution; the textile fibres are optionally subjected during this step to mechanical tension. This operation improves the effectiveness of the fibre dyeing operation, and the fibre tear strength.

A number of possibilities can be envisaged for this operation.

In a first possibility, immersion is carried out with a solution where the concentration of alkaline agent is of the order of 200 to 300 g/l. In a variation, the step is carried out at a temperature of the order of 15°C to 25°C.

In a further variation, it is possible to carry out immersion with a solution with an alkaline agent concentration of about 200 g/l, knowing that the operation is carried out at a temperature in the range 55°C to 70°C, followed by a cooling step and a rinsing step at ambient temperature, placing the textile fibres under tension.

It is also possible to carry out this step with a solution wherein the alkaline agent concentration is 100 – 160 g/l, exerting no tension on the textile fibres, and at a temperature in the range about 20°C to 40°C. Such a case is termed “slack mercerisation” rather than “mercerisation”.

The alkaline solution can also comprise the terpene compound, more particularly in a concentration in the range 1 to 5 g/l.

The textile fibres are then dyed in a series of steps e). These steps are well known in the art.

Dispersed dyes are used, i.e., using pigments which are insoluble in water, or reactive dyes can be used, i.e., using hydrosoluble pigments which fix onto reactive sites present on the fibres.

As an example, and when the fibres are a mixture of synthetic and natural fibres (for example polyester/cotton), dyeing can comprise the following steps: colouring the synthetic fibres (step e1)), fixing these pigments (step e2)), flushing out the non fixed dyes (step e3)), colouring the natural fibres (step e4), washing the non fixed pigments (step e5)).

When the fibres comprise only one or other type of fibre, then only the steps which are specific for this type of fibre are carried out.

Preferably, the dyeing operations are carried out at a pH of at least 7, preferably at least 8.

Such pHs are obtained by using alkaline compounds such as alkali metal hydroxides.

The solutions or dispersions used are aqueous.

In addition to pigments or dyes, the solutions or dispersions comprise a variety of surfactants, which are preferably anionic or non ionic. Examples which can be used are fatty alcohol sulphate ethers, which may be alkoxyated (ethoxylated and/or propoxylated), phosphoric acid esters which may be alkoxyated, mono- and di-alkylsulphosuccinates, polyalkoxyated fatty alcohols, lignosulphonates, the condensation products of formaldehyde and aromatic sulphonic acids (naphthalene sulphonic acid, for example), etc..

It is also possible to use the following as dye liquor additives: colloids such as polysulphonates, polycarboxylates (polyacrylate, acrylic acid/maleic acid copolymer), polyvinylsulphonates, alginates, polysaccharides, or cellulose derivatives.

These surfactants and colloids stabilise the dispersion but can also prevent impurities from precipitating or agglomerating in the liquor.

The solutions or dispersions can also comprise the terpene compounds. They are preferably non ionic.

The use of terpene compounds as defined above is highly advantageous in the context of the dyeing steps. They are very slightly or non-foaming or can even be foam breakers, even under dyeing conditions, in which shear is very high.

Thus the use of the terpene compounds cited above no longer makes the presence of large quantities of anti-foaming agent necessary. It can even be possible to dispense with the use of such agents.

The amount of these terpene compounds is advantageously in the range 1 to 5 g/l.

The dyeing operations proper are carried out conventionally, in particular in batches (jet or jigger).

They can be carried out continuously or, as is preferable, batchwise.

Dyeing is generally carried out hot, i.e., at temperatures in the range 50°C to 90°C. Higher temperatures can be envisaged if the operation is carried out under pressure.

In continuous processes, the textile fibres are first impregnated in the dye liquor, then passed through rollers to remove the excess liquor. The fibres can undergo several passes before the colours are fixed. High shear conditions are particularly encountered during the impregnation steps, causing foam to appear.

In batch processes carried out in a jigger, the fabric is unrolled from one cartridge to another, with immersion in the liquor in between. In this case, agitation is moderate. For this reason, the amounts of foam created is also moderate. However, the foam may be deposited on the textile fibres and be the cause of appearance of spots.

In jet type batch processes, a lot of foam appears. The fabric is in fact entrained in jets of water. When centrifugal pumps are used, the shear is very high and agitation is very strong. Obligatory conventional anti-foaming agents can be destabilised and cause that agent to be deposited on the fabric, with the consequent appearance of spots.

Once impregnation has been completed, the dyes are fixed on the textile fibres by heat.

When the textile fibres have been dyed, a finishing step can be carried out, with the aim of endowing the textile with particular properties depending on the ultimate intended use.

Thus the textile fibres can be treated with softeners (anionic, cationic, silicone), agents which render the fabric more or less stiff (resins), anti-stain agents (fluorocarbons), or agents which can transform the appearance of the fabric (polyurethanes to which flock can be applied to give the appearance of velvet, for example).

A non limiting example will now be described.

EXAMPLE

The tests described below demonstrate the effect of the terpene compound in a step for desizing polyester textile micro-fibres (fabric) compared with a normal surfactant.

Test conditions:

The desizing bath comprised:

Sodium hydrogen (flakes):	2 g/l
Chelating agent (Neocrystal 70, sold by Nicca)	1 g/l
Desizing agent (terpene compound, or normal surfactant)	1 g/l

The fabric was stretched over a support then a drop of oil (HN100 paraffin mineral oil), esterified oil (rapeseed oil methyl ester) or wax (ethylene bis stearamide) was deposited. The ensemble was then treated at 180°C for 1 minute.

The fabric was then immersed in the desizing bath at 110°C for 20 minutes. The weight ratio of the article to be treated to the desizing bath was 1 to 20.

The fabric was then rinsed with an excess of water for 30 seconds, and drained for 30 seconds. The resulting fabric was then dyed with a disperse dye (1% by weight Palanil Violet 3RL – BASF). The article was immersed in the bath comprising the colorant at ambient temperature, and the temperature was then increased to 80°C, with stirring.

The regions in which oil that been deposited had not been removed had been dyed in different manners.

The following desizing agents were tested:

Comparative test I: Igepal NP8.5, a nonylphenol containing 8.5 moles of ethylene;

Test in accordance with the invention: Compound corresponding to cycle 4), the 3- carbon carrying two methyl radicals, and in which X represented $-\text{CH}_2-\text{CH}_2-\text{O}-$, and containing 5 moles of propylene oxide and 9 moles of ethylene oxide.

Comparative test 2: Corresponded to compositions that were free of desizing agents.

Desizing test (removal of oil stain)

The results were graded from 1 (very poor) to 7 (excellent).

Extraction test following desizing

A portion of the treated fabrics was used to measure the percentage of residues (oil, desizing agent) remaining on the fabric.

The residue was extracted with ethyl ether.

The residue corresponded to 100 x the dry extract weight/weight of fabric piece.

For this measurement, the desizing bath contained 3 g/l of desizing agent.

Measurement of foam height

The desizing bath employed was that used in test 1.

The foam was obtained by stirring 900 g of solution with a turbomixer (40 mm turbine, 2000 rpm) for 5 minutes. The temperature was 50°C.

The height of the foam was recorded in a sample after leaving to stand for 5 minutes.

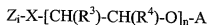
The results are shown in the following table:

Test	Oil	Comparative test 1	Comparative test 2	Invention
Test 1	Mineral	7	2	7
Test 1	Esterified	4	2	4
Test 1	Wax	5	2	5
Test 2	(%)	0.8	1.44	0.7
Foam	(ml)	> 500	< 100	< 100

It can be seen that the agent in accordance with the invention was as effective as the agent of comparative example 1 as regards desizing, and surprisingly as regards the amount of residue remaining on the fibres after treatment. In contrast, the desizing agent of the invention performed better than the agent of comparative example 1 as regards the foam height.

CLAIMS

1. A process for treating textile fibres comprising at least one of the following steps: a) singeing, b) desizing, c) cleaning, d) bleaching, e) mercerisation, f) dyeing steps, g) finishing, characterized in that a compound with formula (I) is used in at least one of the above steps:



where

- Z_1 represents a bicyclo[a,b,c]heptenyl radical or a bicyclo[a,b,c]heptyl radical, optionally substituted by at least one C_1 - C_6 alkyl radical, preferably methyl;

a, b and c being such that

- $a + b + c = 5$;
- $a = 2, 3$ or 4 ;
- $b = 1$ or 2 ;
- $c = 0$ or 1 ;

and comprising a skeleton selected from those shown below (Z_1 to Z_7), and from their corresponding heptyls with no double bond:

1)



[3.2.0]

2)



[3.2.0]

3)



[2.2.1]

4)



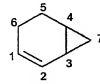
[3.1.1]

5)



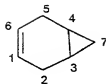
[3.1.1]

6)



[4.1.0]

7)



[4.1.0]

- X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ or $-\text{O}-\text{CH}(\text{R}^{1'})-\text{CH}(\text{R}^{2'})-\text{O}-$ in which:
 - R^1 and R^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1-C_6 hydrocarbon radical;
 - $\text{R}^{1'}$ and $\text{R}^{2'}$, which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1-C_{22} hydrocarbon radical;
- R^3 and R^4 , which may be identical or different, represent a hydrogen atom or a linear or branched C_1-C_{22} (cyclo)alkyl or (cyclo)alkenyl group;
- n is an average value in the range 1 to 200;
- A represents a hydrogen atom, a C_1-C_6 alkyl radical, an aryl or alkylaryl radical, a halogen atom, a $-\text{CH}_2-\text{CH}(\text{OH})\text{R}^5$ group where R_5 represents a linear or branched or cyclic C_1-C_{22} alkyl radical or an aryl radical, or a group selected from $-\text{SO}_3\text{M}$, $-\text{OPO}_3(\text{M})_2$, $-(\text{CH}_2)_a-\text{COOM}$, $-(\text{CH}_2)_b-\text{SO}_3\text{M}$, where a and b are from 1 to 6, M representing H, Na, K, Li, $\text{N}(\text{R}_4)^+$ where radicals R, which may be identical or different,

represent a hydrogen atom or a linear or branched or cyclic C₁-C₂₂ alkyl radical which may be hydroxylated;

or a mixture of these compounds.

2. A process according to the preceding claims, characterized in that a compound with formula
 5 (I) is used wherein radical Z_i is preferably attached to the chain residue via any one of its carbon atoms 1 to 6, and more particularly via carbon atoms 1, 5 and 6.
3. A process according to any one of the preceding claims, characterized in that the compound is such that in formula (I), radical Z is substituted by two C₁-C₆ alkyl groups, on at least one of its carbon atoms, more particularly on carbon atom 7.
- 10 4. A process according to any one of the preceding claims, characterized in that a compound with formula (I) is used wherein X represents -CH₂-C(R¹)(R²)-O- and radicals Z_i correspond to radicals Z₃ to Z₇, preferably to radicals Z₄ and Z₅.
5. A process according to any one of the preceding claims, characterized in that a compound with formula (I) is used in which X represents -O-CH(R¹)-CH(R²)-O- , radical Z_i
 15 corresponds to radical Z₃ not comprising an ethylenically unsaturated bond.
6. A process according to the preceding claims, characterized in that the compound is such that in formula (I), radical Z is substituted by a C₁-C₆ alkyl radical, preferably a methyl radical on carbon 2 or 5 of the bi-cycle.
7. A process according to any one of the preceding claims, characterized in that a mixture of
 20 compounds with formula (I) is used in which at least one thereof is such that the group -[CH(R³)-CH(R⁴)-O]-corresponds to the following sequence: -[CH(R³)-CH(R⁴)-O]_p-[CH₂-CH₂-O]_q-[CH(R³)-CH(R⁴)-O]_r-, where R³ or R⁴ is other than hydrogen, and n=p+q+r.
8. A process according to any one of the preceding claims, characterized in that the compound with formula (I) is used during dyeing steps e).

9. A process according to any one of the preceding claims, characterized in that the compound with formula (I) is used in a concentration in the range 1 to 5 g/l.

**COMBINED DECLARATION AND POWER OF ATTORNEY
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

USE OF POLYALKOXYLATED TERPENE DERIVATIVES FOR TREATING TEXTILE FIBRES

the specification of which (check only one item below):

- ☐ is attached hereto.
- ☐ was filed as United States application
Number _____ on _____
and was amended _____ on _____ (if applicable).
- ☒ was filed as PCT international application
Number PCT/FR00/01543 on 6 JUNE 2000
and was amended _____ on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:				
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365	
France	99/07240	7 June 1999	x Yes	No
			Yes	No
			Yes	No
			Yes	No
			Yes	No

**COMBINED DECLARATION AND POWER OF ATTORNEY
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

USE OF POLYALKOXYLATED TERPENE DERIVATIVES FOR TREATING TEXTILE FIBRES

the specification of which (check only one item below):

- ☐ is attached hereto.
- ☐ was filed as United States application
Number _____ on _____
and was amended _____ on _____ (if applicable).
- ☒ was filed as PCT international application
Number PCT/FR00/01543 on 6 JUNE 2000
and was amended _____ on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:				
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365	
France	99/07240	7 June 1999	x Yes	No
			Yes	No
			Yes	No
			Yes	No
			Yes	No

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

William L. Mathis	17,337	Eric H. Weisblatt	30,505	Bruce T. Wieder	33,815
Robert S. Swecker	19,885	James W. Peterson	26,057	Todd R. Walters	34,040
Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Baum	36,086
Ronald L. Grudziecki	24,570	T. Gene Dillahunty	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopecski	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Slitter	26,999	William H. Benz	25,952	Wendi L. Weinstein	34,456
Samuel C. Miller, III	27,360	Peter K. Skiff	31,917	Mary Ann Dillahunty	34,576
Robert G. Mukai	28,531	Richard J. McGrath	29,195	Donna M. Meuth	36,607
George A. Ilovancec, Jr.	28,223	Matthew L. Schneider	32,814	Mark R. Kresloff	42,766
James A. LaBarre	28,632	Michael G. Savage	32,596		
E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Denny Huntington	27,903	Charles F. Wieland III	33,096		



21839

and:

Address all correspondence to:

Norman H. Stepno
BURNS, DOANE, SWECKER & MATHIS, L.L.P.
P. O. Box 1404
Alexandria, Virginia 22313-1404



21839

Address all telephone calls to: 703-836-6620 at (703) 836-6620.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST INVENTOR	Gilles LORENTZ
Signature	<i>[Signature]</i>
Date	5 February 2002
Residence (City, State, Country)	Lyon, France FR
Citizenship	French
Mailing Address	129, rue Vauban
City, State, ZIP, Country	F-69006 Lyon, FRANCE
FULL NAME SECOND INVENTOR, IF ANY	André VAN DER SPUY
Signature	
Date	
Residence (City, State, Country)	Ribeauvillé, France
Citizenship	French
Mailing Address	9A, rue Henri Kugler
City, State, ZIP, Country	F-68150 Ribeauvillé, FRANCE

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

William L. Mathis	17,337	Eric H. Weisblatt	30,505	Bruce T. Wieder	33,815
Robert S. Swecker	19,885	James W. Peterson	26,057	Todd R. Walters	34,040
Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Baum	36,086
Ronald L. Gruzdzicki	24,570	T. Gene Dillahunty	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopeccki	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Sluiter	26,999	William H. Benz	25,952	Wendy L. Weinstein	34,456
Samuel C. Miller, III	27,360	Peter K. Skiff	31,917	Mary Ann Dillahunty	34,576
Robert G. Mukai	28,531	Richard J. McGrath	29,195	Donna M. Meuth	36,607
George A. Hovanec, Jr.	28,223	Matthew L. Schneider	32,814	Mark R. Kresloff	42,766
James A. LaBarre	28,632	Michael G. Savage	32,596		
E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Danny Huntington	27,903	Charles F. Wieland III	33,096		



21839

and:

Address all correspondence to:



21839

Norman H. Stepno
BURNS, DOANE, SWECKER & MATHIS, L.L.P.
P.O. Box 1404
Alexandria, Virginia 22313-1404

Address all telephone calls to: 703-836-6620 at (703) 836-6620.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST INVENTOR	Gilles LORENTZ
Signature	
Date	
Residence (City, State, Country)	Lyon, France
Citizenship	French
Mailing Address	129, rue Vauban
City, State, ZIP, Country	F-69006 Lyon, FRANCE
FULL NAME SECOND INVENTOR, IF ANY	André VAN DER SPUY
Signature	<i>[Signature]</i>
Date	25/12/02
Residence (City, State, Country)	Ribeauvillé, France FR
Citizenship	French
Mailing Address	9A, rue Henri Kugler
City, State, ZIP, Country	F-68150 Ribeauvillé, FRANCE